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During this project, GaN has been grown on lithium gallate with much improved structural quality, excellent optical properties and uch improved electrical properties superior nitride properties resulted from a detailed investigation and understanding of the surface chemistry of LGO. As a polar material, LGO has a cation terinated (lithium and gallium face and an anion (oxygen) face. It was determined that nitride films grown on the anion face cracked and peeled (see Figure 3) where as films grown on the cation face are smooth and adherent (see Figure 3). Having identified the proper face for growth and determined the upper limit of substrate temperatures, high quality growth of GaN on LGO was then possible.

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Final Report for:

MBE Grown Nitrides for High Temperature Electronics Using Compliant Substrates

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Motivation for using Lithium Gallate as a Substrate for Gallium Nitride

The growth of gallium nitride (GaN) on lithium gallate (LGO) affords many advantages compared to all other available substrates. Lithium gallate offers the best lattice match of any available substrate. The lattice match of several substrates is summarized in table I [1]. Lithium gallate offers the smallest average lattice mismatch of any available substrate for the III-nitrides. Lithium gallate has an orthorhombic crystal structure as indicated in figure 1. Thus, lithium gallate has different a-axis and b-axis lattice constants. When GaN is grown on LGO, a different lattice match occurs in the a-axis direction verses b-axis direction (see figure 1). The mismatch in the b-axis direction is -0.19% whereas the mismatch in the a-axis direction is +1.9%. This results in an average mismatch of only +0.86%. Our in depth x-ray diffraction studies have indicated that the GaN film actually rotates slightly about the c-axis to further minimize the mismatch. Unlike other substrates with larger lattice mismatches, elastically strained GaN can be grown on LGO to much greater thickness' (known as the critical thickness) before the generation of dislocations occurs.

One important consideration of lattice match that is often overlooked is the c-axis lattice match. Materials such as 6H SiC have large c-axis lattice constants (15.1173 Å) compared to GaN which is 5.185 Å. This can lead to stacking faults at step edges. LGO has a c-axis lattice constant of 5.012 Å resulting in an excellent c-axis lattice match of 3.3%.

When GaN is grown on substrates with very large lattice mismatch such as sapphire, the GaN minimizes it's strain by tilting slightly as indicated in figure 2. Each individual grain of GaN can be oriented in a different direction. This leads to low angle grain boundaries and mosaic spread in the c-axis of individual grains. Carriers traveling laterally from one grain to another must be scattered into the conduction paths of the new grain. This results in lowered mobility. Low lattice

mismatch materials such as LGO, ZnO and SiC do not suffer from large mosaic spread. The lack of mosaic structure means that truly single crystal material can be grown on these low mismatch substrates. The term "single crystal" can be accurately used due to the identical orientation of adjacent grains across the dislocation array (see figure 2). The lower the lattice mismatch, the better the grain to grain alignment. The resulting zero angle grain boundaries (more appropriately termed dislocations arrays) should lead to higher mobility of carriers in the lateral direction. This increased mobility will strongly impact devices such as FET's where lateral current conduction dominates.

Much attention has been placed on the polarity of GaN [2]. Control of the polarity is important for maximizing the piezoelectrically induced charge in the channels of FETs [3], minimizing the inversion domains that can limit mobility [4], and determining the smoothness of surfaces and interfaces [4]. Non-polar materials such as sapphire, and silicon do not inherently nucleate films of a single polarity. Polar materials such as LGO, ZnO and SiC can nucleate GaN of a particular polarity. This is because anions chemically prefer to nucleate on cation layers, resulting in a preference for cation polarity (after the deposition of a single monolayer). LGO is a strongly polar crystal with lithium and gallium (cations) oriented in the same atomic plane while oxygen (the anion) is arranged in the underlying plane. With this atomic arrangement, gallium polarity GaN can be easily grown. Furthermore, using NaOH etches to determine the polarity [2] of our GaN films has indicated a single polarity GaN. Due to the complex chemistry and high vapor pressure of nitrogen on the nitrogen face of GaN, further research is needed to allow the growth of nitrogen polarity GaN. The ability to control the polarity of GaN is a major advantage of LGO and can lead to novel device design.

The two most common substrates used for III-Nitride growth, sapphire an SiC, are very hard and are difficult to etch. This feature prevents the easy removal of the substrates or the backside

patterning of such substrates. Of particular interest are backside patterning for electrical contact, heat pipes, and free standing GaN membranes. LGO is unique in that, not only can it be etched, it can be etched incredibly fast. Our work has indicated that an entire 0.5 mm thick substrate can be chemically wet etched in 10 minutes. Thus, tedious and costly mechanical lapping or plasma etching is not necessary. This extra degree of freedom in device design could allow the development of power devices with heat dissipation pipes or on low cost ceramic heat sinks and/or micromechanical devices that are not practical with harder substrates.

Table I

CRYSTAL	A-AXIS MISMATCH TO GaN %	B-AXIS MISMATCH TO GaN %	C-AXIS MISMATCH TO GaN %	THERMAL EXPANSION (10^{-6} /K)
AlN	-2.1	---		4.2
GaN	---	---	---	4.5
InN	2.3	---		Unknown
$LiAlO_2$	-1.45	---		7.5
$LiGaO_2$	1.9	***-0.19***	3.3	7
Al_2O_3	8.7	---		7.5
ZnO	2.26	---		2.9
6H-SiC	3.14	---	374.6% (–2.8% ignoring stacking)	

Values for a-axis taken from [1]. Values for b and c-axes are calculated.

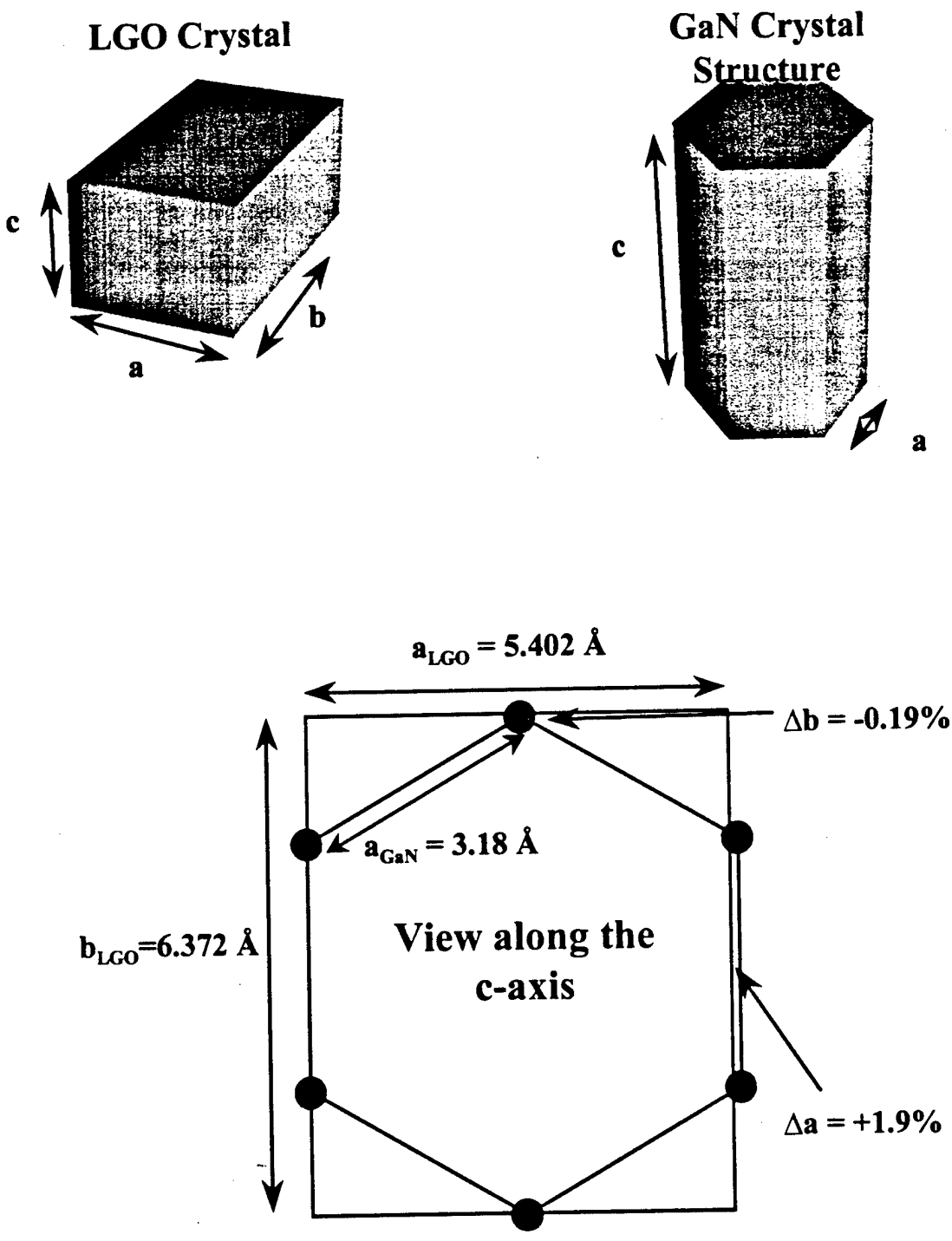
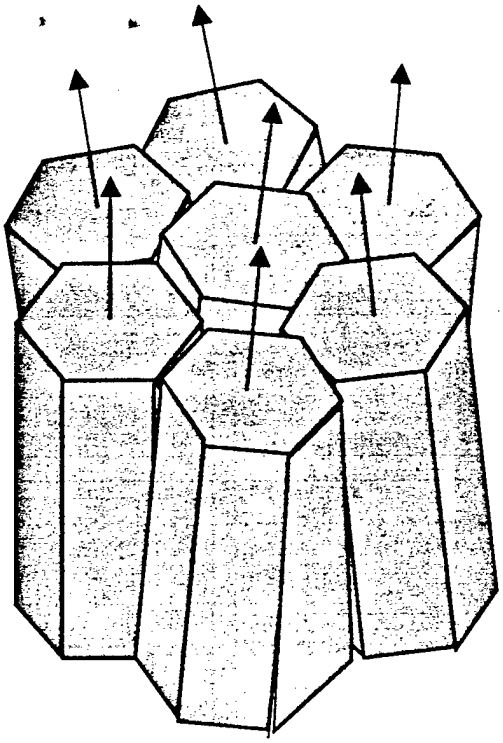
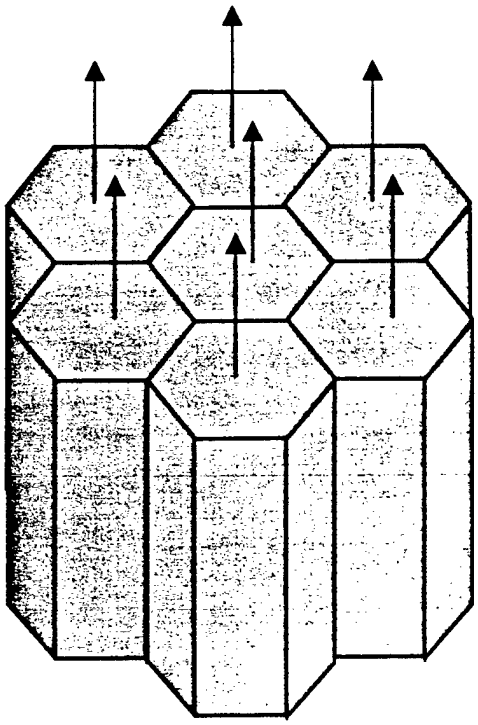

LGO Crystal
c
b
a
GaN Crystal Structure
c
a
a_{LGO} = 5.402 Å
Δb = -0.19%
a_{GaN} = 3.18 Å
b_{LGO}=6.372 Å
View along the c-axis
Δa = +1.9%

Figure 1



Mosaic Structure due to large lattice mismatch: each grain is misaligned



No Mosaic Structure resulting from small lattice mismatch: each grain is aligned

Figure 2

High Quality Gallium Nitride Grown on Lithium Gallate

A compliant substrate is, by design, very thin and must be of the highest quality possible. Most material grown on other substrates is too thick to be used as a compliant substrate and of poor quality. Because of the ability to grow very thin, elastically strained GaN on LGO, and it's rapid etch rate (the entire substrate can be chemically removed in ~five to ten minutes) for substrate removal, LGO is a near ideal substrate for development of a compliant nitride substrate. Throughout this program, very high structural quality, extremely thin GaN has been developed using LGO substrates and plasma assisted molecular beam epitaxy.

During this project, GaN has been grown on lithium gallate with much improved structural quality, excellent optical properties and much improved electrical properties. These superior nitride properties resulted from a detailed investigation and understanding of the surface chemistry of LGO. As a polar material, LGO has a cation terminated (lithium and gallium) face and an anion (oxygen) face. It was determined that nitride films grown on the anion face cracked and peeled (see Figure 3) where as films grown on the cation face are smooth and adherent (see Figure 3). Furthermore, desorption mass spectroscopy investigations (see Figure 4) indicated that at substrate temperatures greater than ~ 850 degrees C, the substrate becomes unstable, and looses both lithium and gallium. X-ray photoelectron spectroscopy indicates that the remaining surface is lithium rich. This thermal instability explains why previous attempts at growth at elevated temperatures resulted in poor results. Having identified the proper face for growth and determined the upper limit of substrate temperatures, high quality growth of GaN on LGO was then possible. Smooth, reconstructed surfaces, as shown in Figure 5, result during growth. These smooth, gallium terminated (as determined by etch studies) films result in

excellent structural quality very thin films.

As shown in Figure 6, very thin films, <0.3 μm thick, have resulted in <0004> X-ray diffraction rocking curves of 145 arc-seconds, and <10-15> of 245 arc seconds. The small asymmetric rocking curve indicates that the small symmetric FWHM is not the result of columnar growth of GaN. Rather, the small symmetric FWHM results from the excellent lattice match of GaN to LGO. Thicker films, ~1 μm thick, have resulted in 85 arc-seconds <0004> reflections (see Figure 7). The X-ray reciprocal space maps, like that shown in Figure 8, have indicated that the nitride film is epitaxially aligned with the substrate and show very little dispersion. The films grown at ~ 690 degrees C indicate elastically strained GaN where as higher substrate temperatures indicated no elastic strain.

TEM imaging has confirmed the epitaxial relationship of the nitride films and has revealed small numbers of threading dislocations observed in cross section (see Figure 9). The plane view TEM image shown in Figure 10

indicates a dislocation density of ~6e8 cm^{-2}, among the best found for pure heteroepitaxy of GaN.

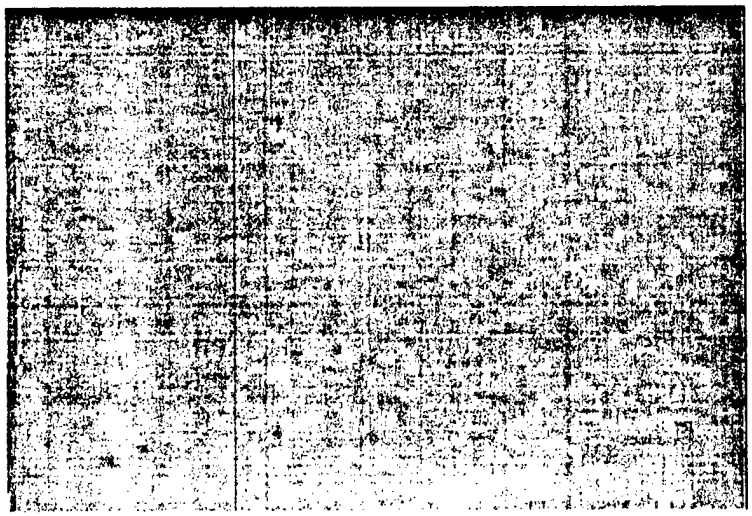
As indicated in Figure 12, comparisons of heterostructures consisting of $Al_{0.26}Ga_{0.74}N$ grown on GaN indicate a 5 fold improvement in the X-ray line widths of the aluminum gallium nitride layer when the structure is grown on LGO instead of sapphire. This, in turn, results in improved 2 dimensional FET structure mobilities. Two dimensional heterostructure mobilities of 730 cm^2/V-s at room temperature and sheet charge densities of 1.5e13 cm^{-2} have also been achieved using AlGaN/GaN structures. Two dimensional heterostructure mobilities of 2200 cm^2/V-s have been achieved at liquid nitrogen temperatures.

The electrical properties of the GaN bulk films are also much improved. High resistivity (above our measurement limit) undoped films are routinely achieved. Bulk GaN electron mobilities have increased to ~160 cm^2/V-s at silicon doping levels of ~1e18 cm^{-3} . These bulk mobilities may be limited by high lithium impurity concentrations near the LGO / GaN interface. GaN grown on LGO can also be doped p-type using Mg. Currently, the limits of doping are not known, but levels as high as 1e17 cm^{-3} have been achieved. The low growth temperatures used with LGO should prove helpful in achieving reproducibly high hole concentrations.

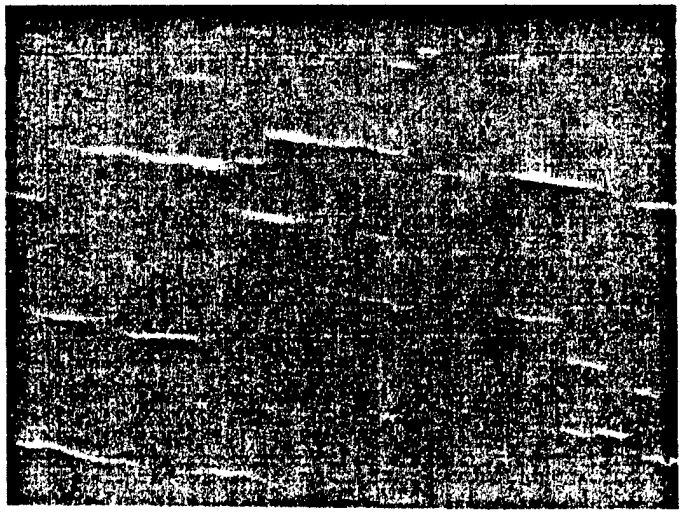
The optical characteristics of the GaN grown on LGO are superb. Figure 13 shows strong UV photoluminescence in GaN grown on LGO. Yellow light is also detected from the sample but was found to primarily be the result of fluorescence from the LGO substrate itself, NOT luminescence from the GaN film. Figure 14 shows the fluorescence of the bare LGO. Undoped films show weaker UV luminescence, indicating the presence of a deep level that is compensated by the n-type silicon dopants. This deep level is most likely related to Li, C or O,

since these elements were found via SIMs analysis to be present in the GaN films at moderate concentrations.

Several attempts have been made to remove GaN films from the LGO to form the GaN template for regrowth (i.e. the compliant substrate). To date, only small area films (~1x3 mm) have been successful. Both epitaxial lift off and bonded substrate removal has been attempted. The limitation on size has been a result of the lack of adhesion of the GaN layer to the host material. Once the substrate is removed, the bond from the GaN to the host must be able to withstand all the "grown in" stress in the film. Typical bonds used, oxide, nitride, polymer, and metal bonds have, thus far, been unable to prevent relaxation of the nitride film. Current efforts are targeted at increasing the adhesion of the bonding layer to the nitride and attempting to reduce the strain between the LGO and nitride film.



GaN film grown on the cation face: Smooth and adherent



GaN film grown on the anion face: cracks and peels

Figure 3

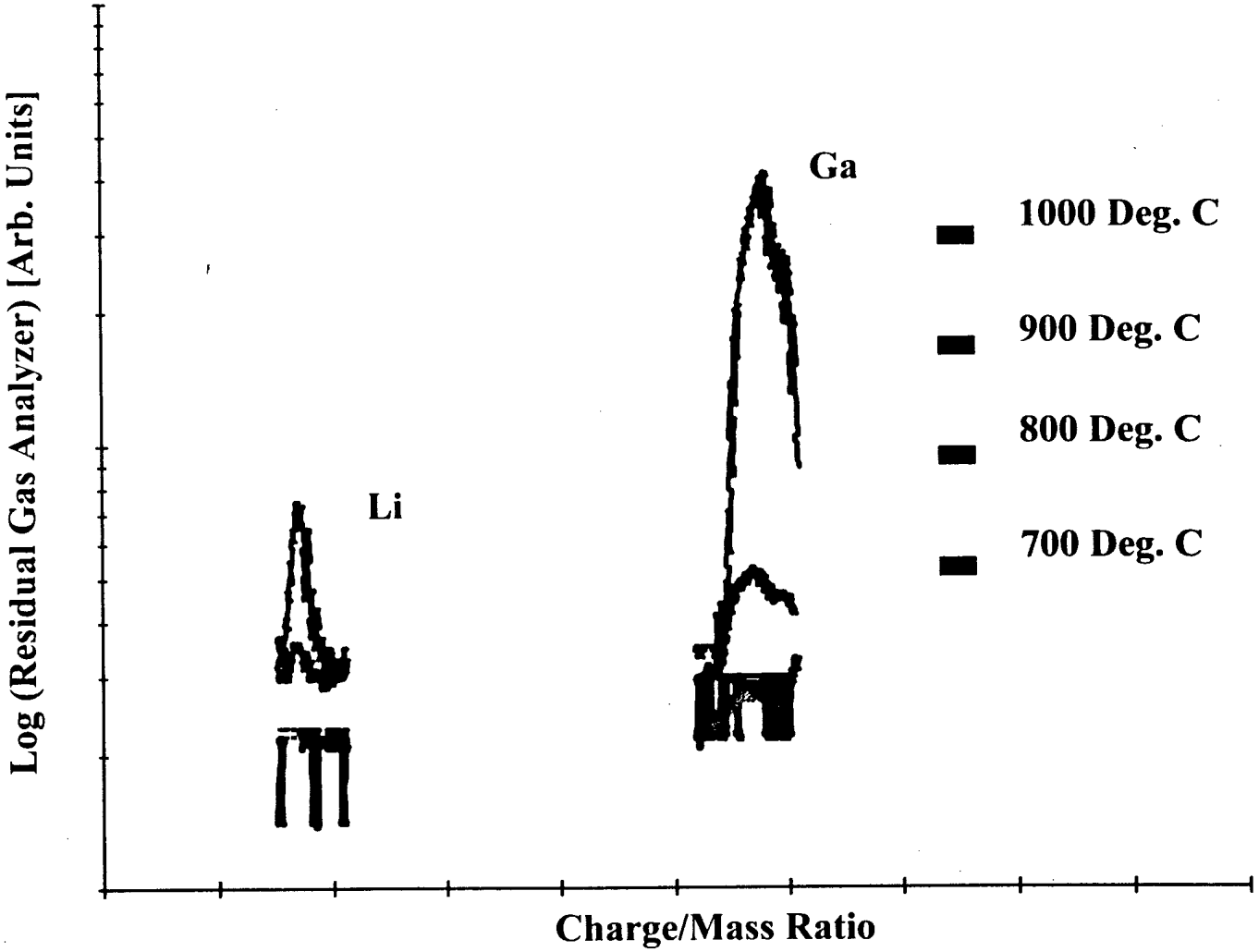

Log (Residual Gas Analyzer) [Arb. Units]
Li
Ga
1000 Deg. C
900 Deg. C
800 Deg. C
700 Deg. C
Charge/Mass Ratio

Figure 4

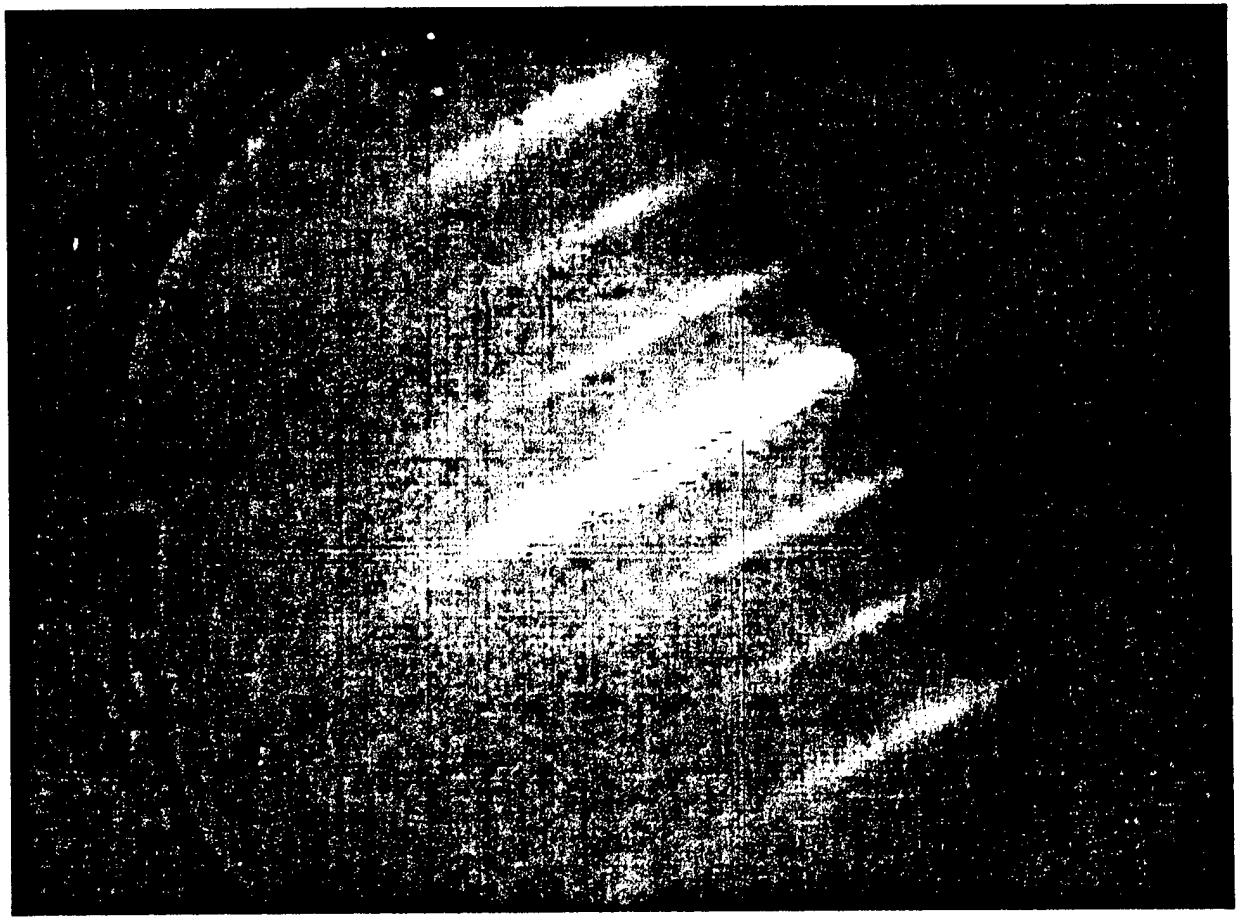


Figure 5

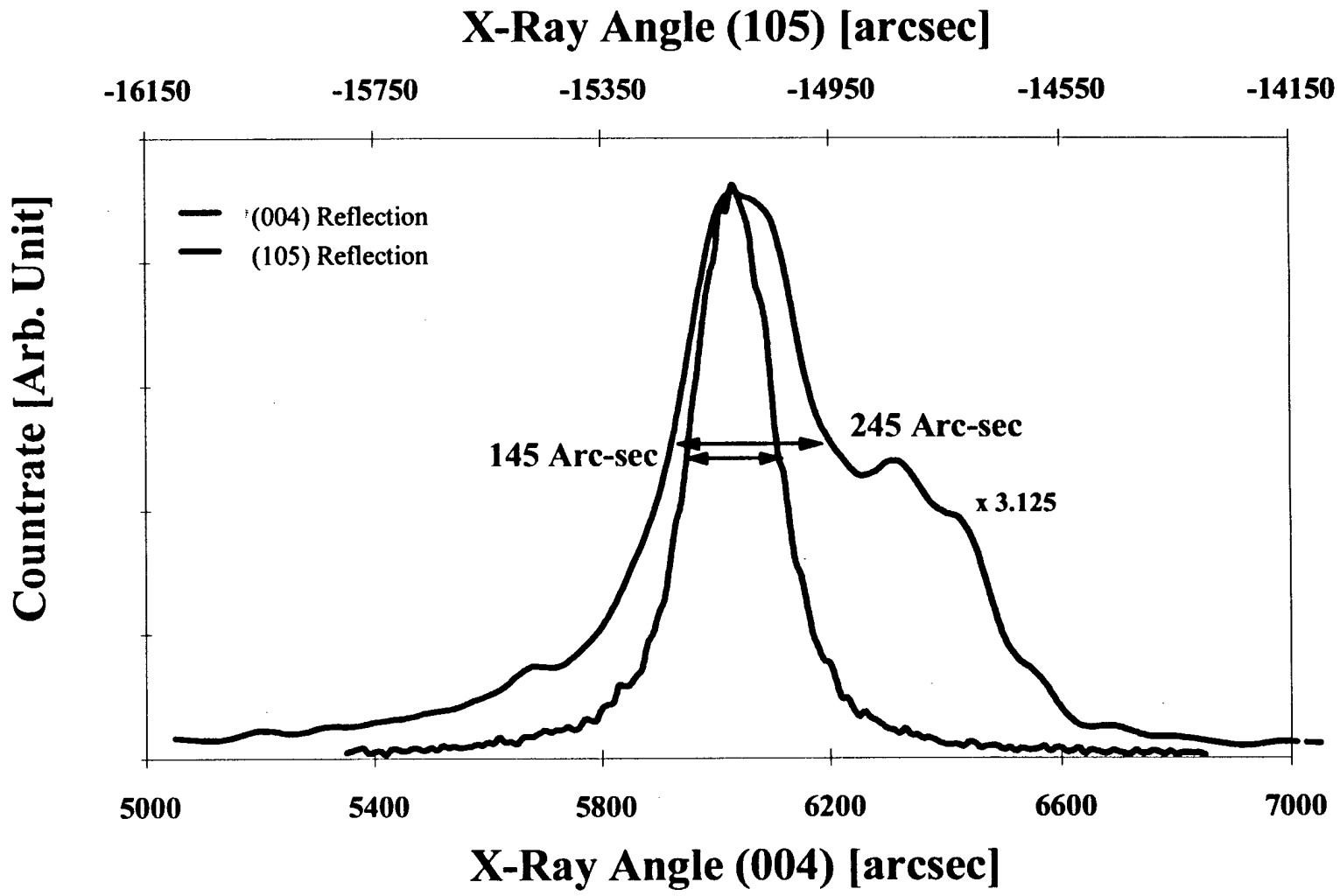

X-Ray Angle (105) [arcsec]
-16150
-15750
-15350
-14950
-14550
-14150
Countrate [Arb. Unit]
(004) Reflection
(105) Reflection
145 Arc-sec
245 Arc-sec
x 3.125
5000
5400
5800
6200
6600
7000
X-Ray Angle (004) [arcsec]

Figure 6

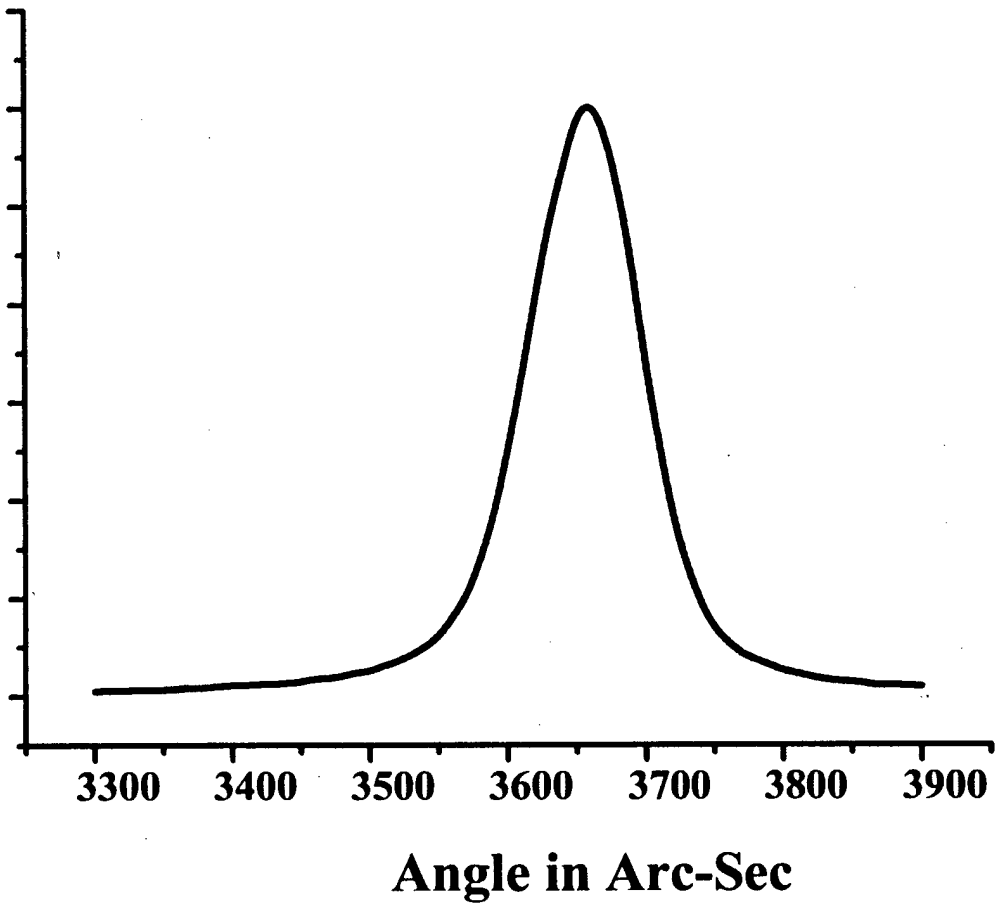


Figure 7

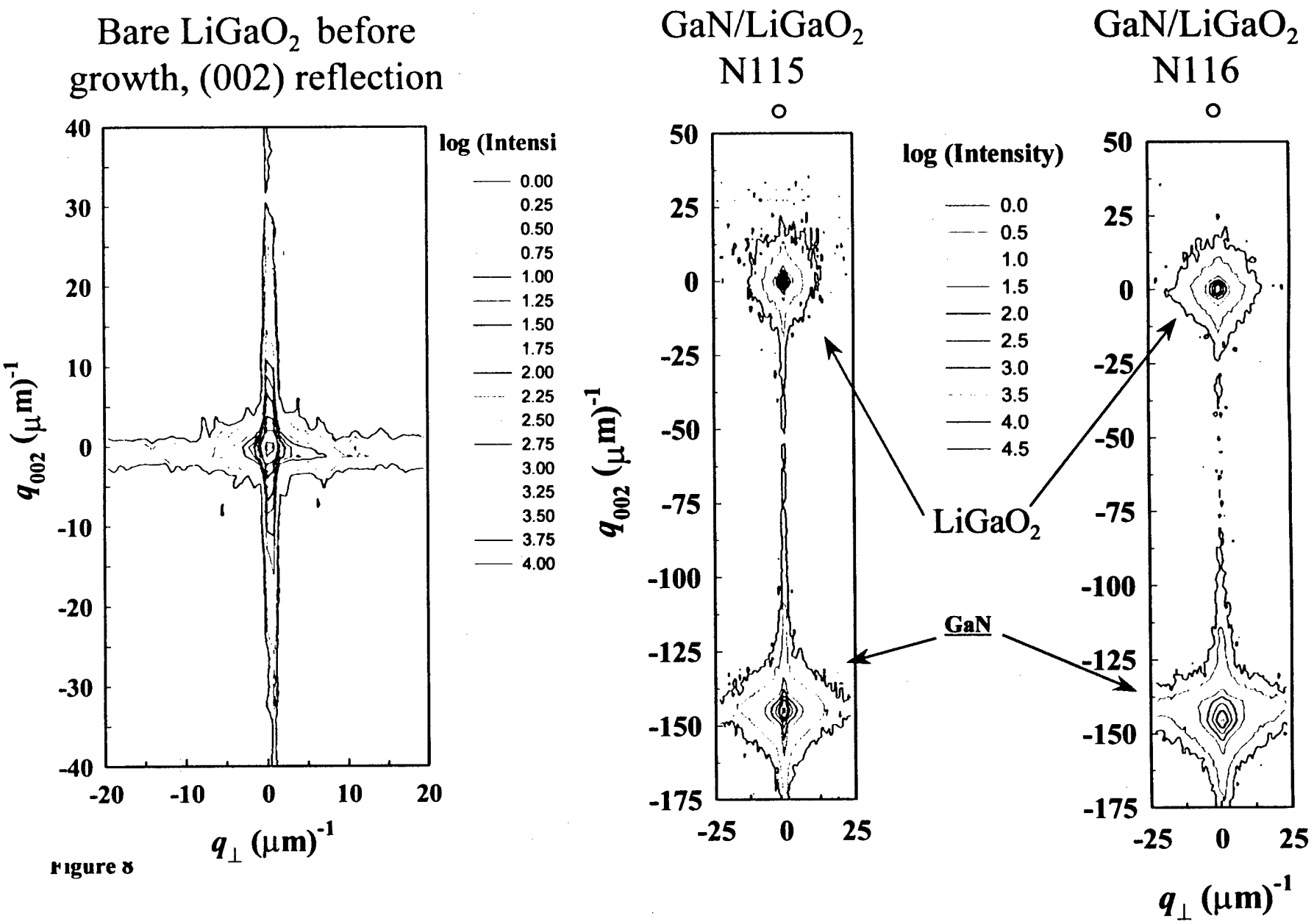

Bare $LiGaO_2$ before growth, (002) reflection
q_{002} $(\mu m)^{-1}$
$q_\perp$ $(\mu m)^{-1}$
log (Intensi
0.00
0.25
0.50
0.75
1.00
1.25
1.50
1.75
2.00
2.25
2.50
2.75
3.00
3.25
3.50
3.75
4.00
$GaN/LiGaO_2$ N115
$GaN/LiGaO_2$ N116
log (Intensity)
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
$LiGaO_2$
GaN

Figure 8

Cross Sectional View

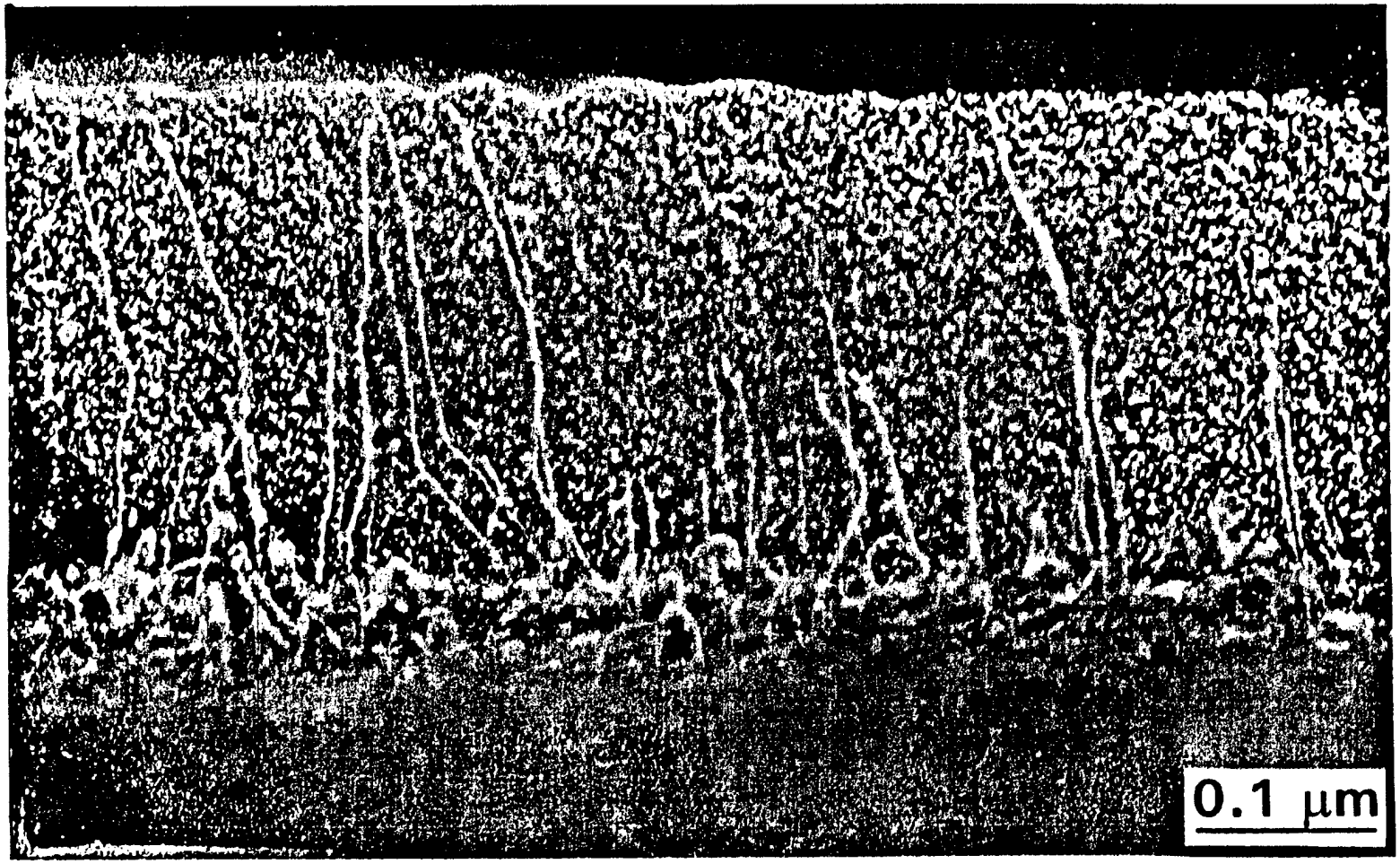


Figure 9

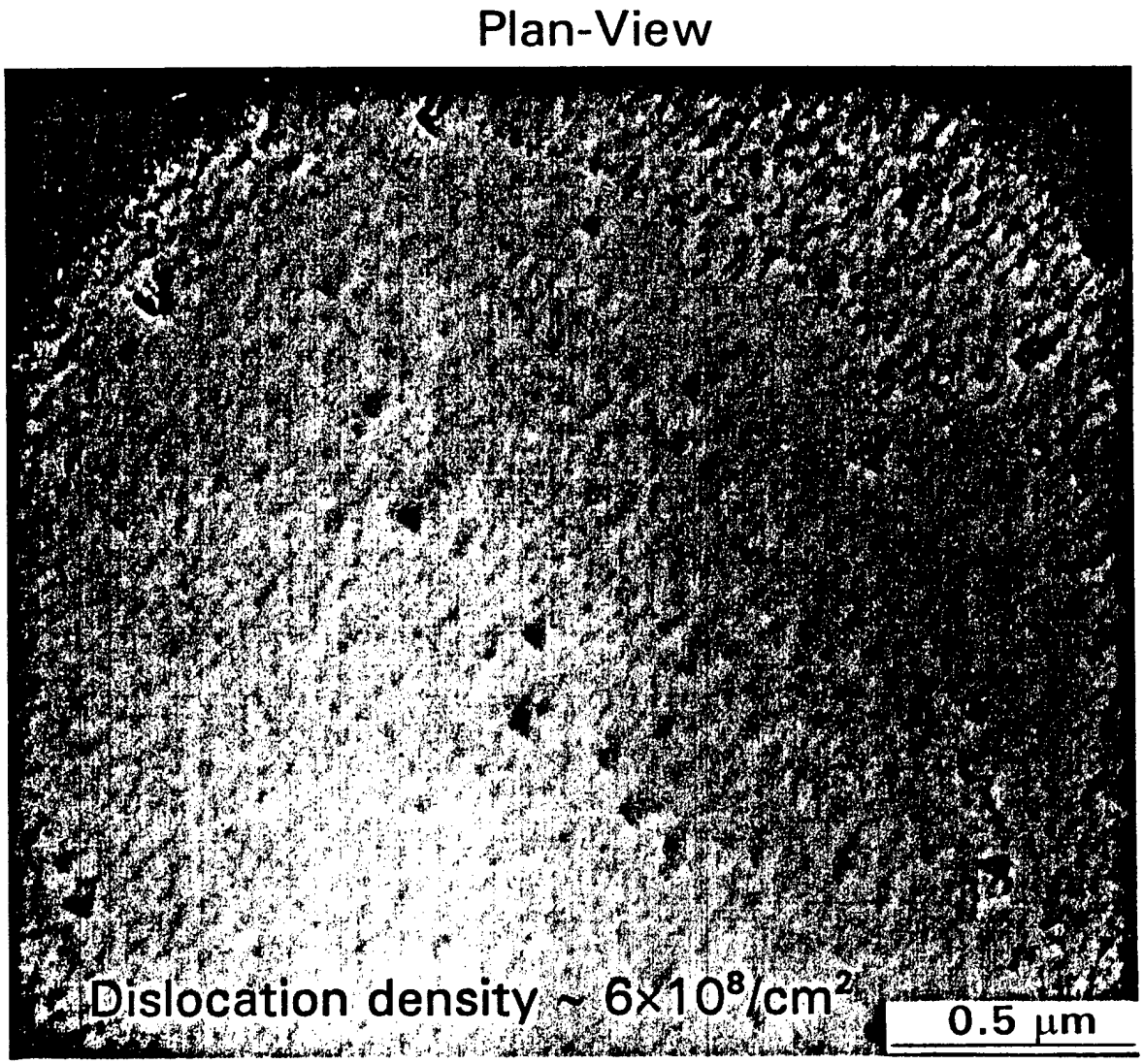


Figure 10

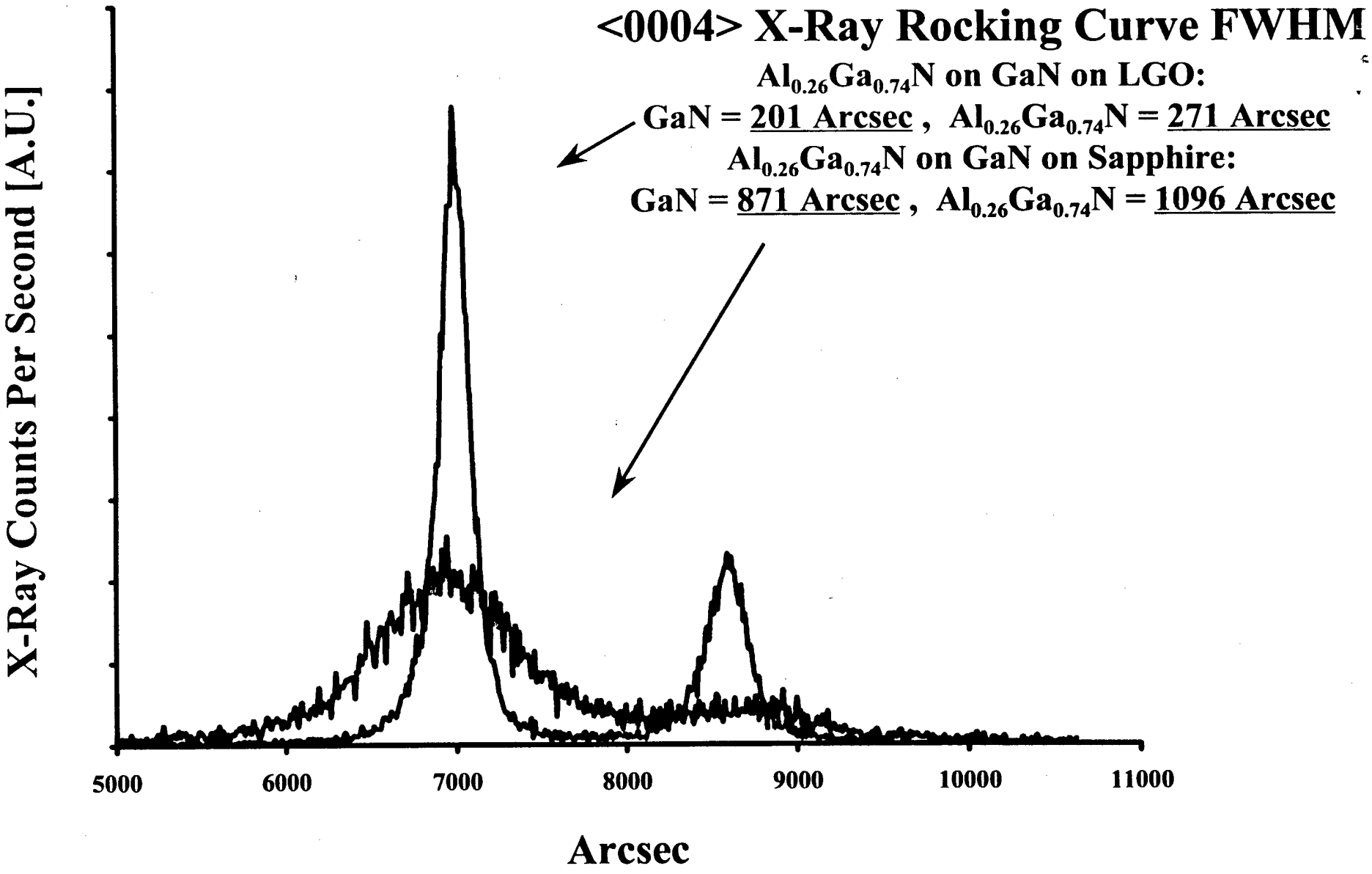

<0004> X-Ray Rocking Curve FWHM
$Al_{0.26}Ga_{0.74}N$ on GaN on LGO:
GaN = 201 Arcsec , $Al_{0.26}Ga_{0.74}N$ = 271 Arcsec
$Al_{0.26}Ga_{0.74}N$ on GaN on Sapphire:
GaN = 871 Arcsec , $Al_{0.26}Ga_{0.74}N$ = 1096 Arcsec
X-Ray Counts Per Second [A.U.]
5000
6000
7000
8000
9000
10000
11000
Arcsec

Figure 12

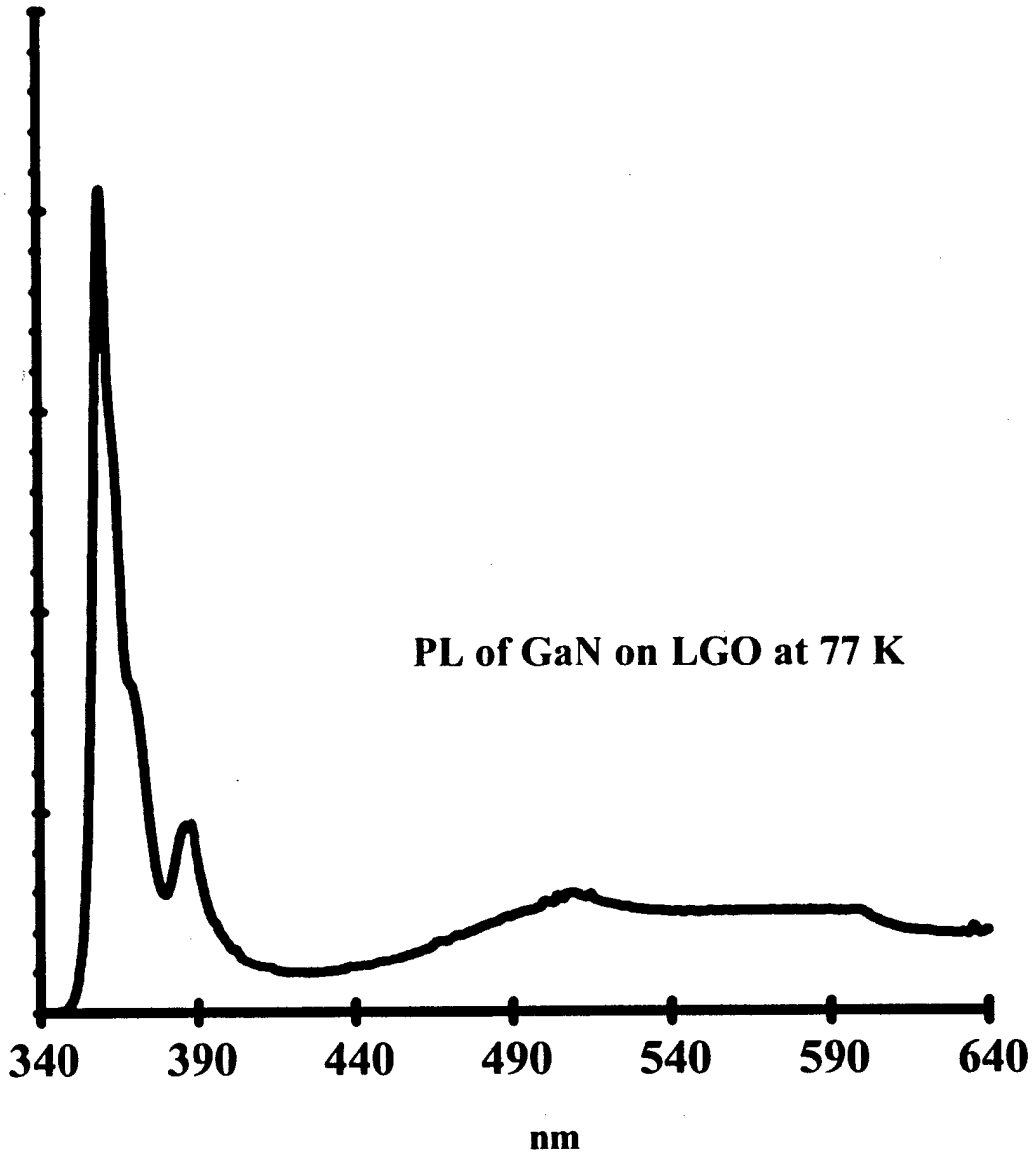


Figure 13

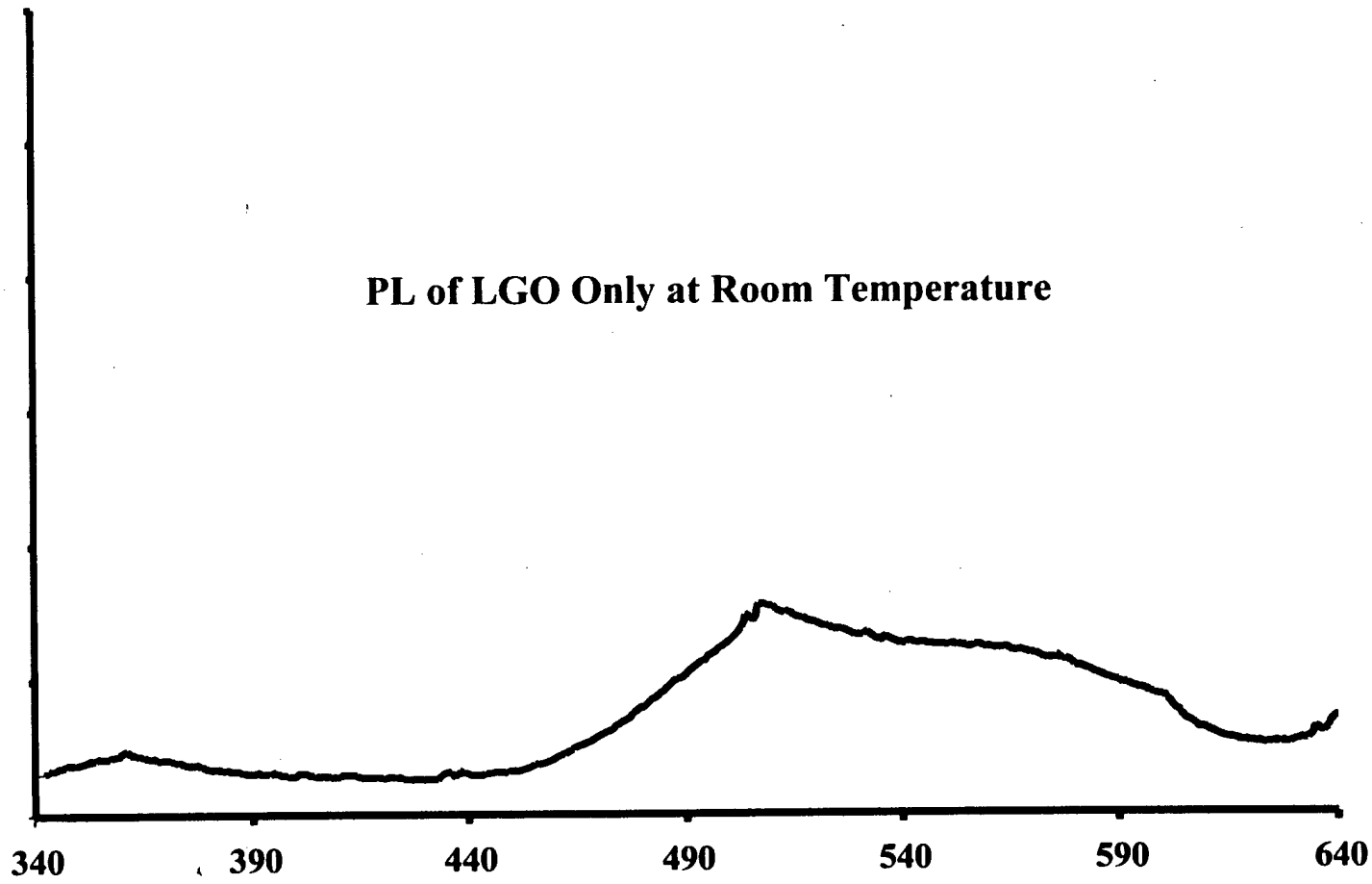


Figure 14

Design of Experiments Approach to Buffer Optimization of Gallium Nitride

The growth of gallium nitride on other more conventional substrates, like sapphire, requires some sort of nucleation process to allow the films to adhere. The complex nature of the nitridization and low temperature buffer combinations possible, result in a vast, multidimensional parameter space that is difficult to optimize. Thus, we have pursued a statistical design of experiment approach to solve this problem. In this approach, many parameters are varied simultaneously, allowing the full breadth of the parameter space to be explored. The advantages of this approach include obtaining a maximum of statistically valid information from a minimum data set, information on the interaction of parameters, the ability to identify system drift and hidden variables, and the ability to determine the statistically optimal nucleation layer. Of most significant is correlations between the post growth surface reconstruction and material quality.

While the theoretical and experimental interpretation of surface reconstruction in cubic-GaN growth is widely accepted, the mechanism leading to this phenomena in hexagonal GaN growth has rather rare and controversial. Herein, we report the correlation between the initial growth conditions and the surface reconstruction after epitaxial growth. The comparison of wurtzite GaN films with and without surface reconstruction is presented based on Hall measurement, photoluminescence and X-ray rocking curve measurements.

Film growth experiments were performed in Riber 32P MBE system using radio frequency plasma assisted nitrogen source. For the present study six factors for the initial stage of GaN growth were chosen, which were time and temperature for nitridation condition, buffer growth temperature, Ga cell temperature, growth time, and nitrogen plasma power during buffer growth. The range of

experimental conditions are tabulated in table III. The traditional approach to determining relationships between growth conditions and material properties has rested on the standard experimental approach - varying one factor at a time while holding the others constant. To facilitate locating the appropriate regime where surface reconstructions occur, we employed a statistical experimental design technique called D-optimal method. While traditional one factor at a time methods require 3^6(=216) experiments for 3 level test for each factor, this statistical technique enable us to use only 30 experiments including two center points.

The group III gallium element was supplied by solid source effusion cell. The growth chamber was kept under a nominal base pressure in 10^{-10} Torr range. To separate the influences of bulk growth conditions on final characteristics from those of initial growth factors, we fixed bulk growth conditions. The epilayer growth following initial treatments were conducted with substrate temperature at 770°C, Ga beam equivalent flux 4.4×10^{-7} Torr, Nitrogen flux 2.2×10^{-5} Torr and Nitrogen power 450W. The temperature of the tantalum backed sapphire substrate has been calibrated based on pyrometer reading. Deposition rates were typically 0.34μm/h. Total film thickness, as measured using a stylus micro-profilometer, ranged from 0.65μm to 0.75 μm . Although all six factors may affect a profound change in structural, electrical and optical properties, our statistical analysis reveal that nitridation time, nitridation temperature and gallium flux during the low temperature buffer growth were responsible for the vast majority of the quality variations. Dispersion of the measured observables under the same conditions came from the less significant three growth conditions, which were not discussed here to avoid the complex issues associated with the simultaneous change in the six initial conditions and so to clarify the crucial role of the first three factors.

In this work, we observed fairly diverse occurrence of strong surface reconstructions during and just after completion of growth, as well as weak reconstruction at low temperature following cooling down. (2x2), (2x4), and (4x4) reconstructions and (1x1) unreconstructed surfaces were observed during growth interrupts, while (1x3) reconstructions were observed during nitridation. A few samples also show a surface reconstruction at 20~30min after cooling down to substrate temperature at about 200°C ~300°C. Since this MBE system is devoted solely to GaN growth using 7N Nitrogen gas source, we have no discernible source of hydrogen, excluding the possibility of hydrogen impurity related surface reconstruction.

Typical RHEED patterns for unreconstructed and reconstructed hexagonal GaN are shown in figure 14. Figure 14 (a) , and (b) show the typical (2x2) reconstruction patterns along $[2\bar{1}\bar{1}0]$ and $[1\bar{1}00]$ azimuths. These (2x2) reconstructions originate from low Ga flux growth during the low temperature buffer together with short nitridation time and high nitridation temperatures. RHEED from low temperature buffer growth of those reconstructed film were very spotty during buffer growth, suggesting that surface reconstructions are related to inferior nucleation processes.

Also shown in figure 14 (c) and (d) , (4x4) reconstructed patterns at ~250 °C during cooling after termination of growth. These patterns appear to be a slight variation of the (2x2) since this high order transition always followed the preceding (2x2) low order reconstruction.

The unreconstructed (1x1) surface were the most stable ones which lasted throughout the whole growth period without blurring or developing spots on Kikuchi lines. These stable growth fronts were obtained from high Ga flux during the low temperature buffer along with long, low temperature nitridation conditions. After long nitridation times at high temperatures, blurry but distinctive RHEED was readily observed from sapphire substrate. This led us the conclusion that

a good template for the subsequent buffer layer growth was well established during this nitridation step.

Resistivity and Hall mobility measurements were conducted at room temperature (295K) and 77K using the van der Pauw method. The characteristics of total 30 GaN samples show Hall mobilities of 20~240 $cm^2/V.s$ with average n-type carrier concentration of Si doped materials was $2.1x10^{18}$ cm^{-3}. The distribution of Hall mobility as a function of Ga flux is visualized in figure 15. As can be clearly seen from the figure, surface reconstructed films suffer from relatively low mobility. Furthermore, the transition from surface reconstruction and non-reconstruction most likely took place around Ga flux $1.6x10^{-7}$ Torr during the buffer growth.

Figure 16 illustrates the characteristics of 78K photoluminescence collected from all samples studied in this work. 77K donor bound excitonic transition peaks at 3.47eV were obtained from these films using the 325nm HeCd laser as an excitation source. Broad line widths is strong evidence of poor quality material, leading to inferior quality of the films for which reconstructions were observed. Whereas those with surface reconstructions had relatively intense yellow luminescence peaks, no discernable deep level yellow peaks were observed for the most of unreconstructed films that were growing under Ga rich buffer condition.

The structural properties of the films were also compared ex-situ by X-ray double crystal rocking curves. The results are summarized in figure 17. Similar to Hall and photoluminescence observations, this measurement also found to be broader FWHM for reconstructed surfaces, again indicating inferior crystal structure. This fact explains the source of low Hall mobility and high resistivity possibly due to potential fluctuations coming from crystal defects. The difference between this work and the documented results is not obvious. These differences may result from unreported

columnar growth of hexagonal GaN which not only lead the good symmetric X-Ray FWHM but also result in poor electrical properties.

Furthermore, Table II summarizes previously published reports where reconstructions occurred and the reported electrical properties.. Although those reports claimed high quality of their materials based upon photoluminescence and x-ray measurement, we can clearly notice inferior mobility of films with surface reconstructed films. These results of electrical properties are consistent with our experiments discussed in the present study.

In summary, as far as streaky RHEED gives a rough evidence for flat and well-defined surface morphology, it seems difficult to claim the high quality material growth by observing the surface reconstruction. On the contrary, based upon above electrical optical measurements, we conclude that surface reconstruction originate from defective structure for wurtzite GaN growth.

Table II. Experimental initial growth conditions for wurtzite GaN on (0001)

Layer	Factor	Range
Nitridation		
	Temperature	600 ~ 800 °C
	Time	1 ~ 30 min
LT Buffer		
	Growth Temp.	400~600 °C
	Ga Flux (BEP)	$1.0x10^{-7}$ ~ $2.0x10^{-7}$Torr
	RF Plasma Power	350 ~ 500 Watt
	Growth Time	3 ~ 15 min

Table III. Summary of the published experimental data on electrical properties

Reference	Substrate (cm^{-3})	Hall Concentration ($cm^2/V.s$)	Mobility	Remark
Lin et al '93	6H-SiC	~10^{17}	100 ~ 200	
Molar et al '94	Al_2O_3	3.4 x10^{18}	77	
Hughes et al '95	6H-SiC	2 x10^{15}~ 5x10^{17}	unpublished	
Wong et al '96	Al_2O_3	unpublished	50 ~60	
Iwata et al '96	Al_2O_3	9 x10^{17}	unpublished	ECR Ion removed
Hacke et al '96*	Al_2O_3	8.34 x10^{18}	80	(2x2) regime
		5.87 x10^{17}	142	
		7.87 x10^{17}	212	near (1x1) regime
Piquette et al '98	Al_2O_3	5 x10^{17}	50 ~ 80	

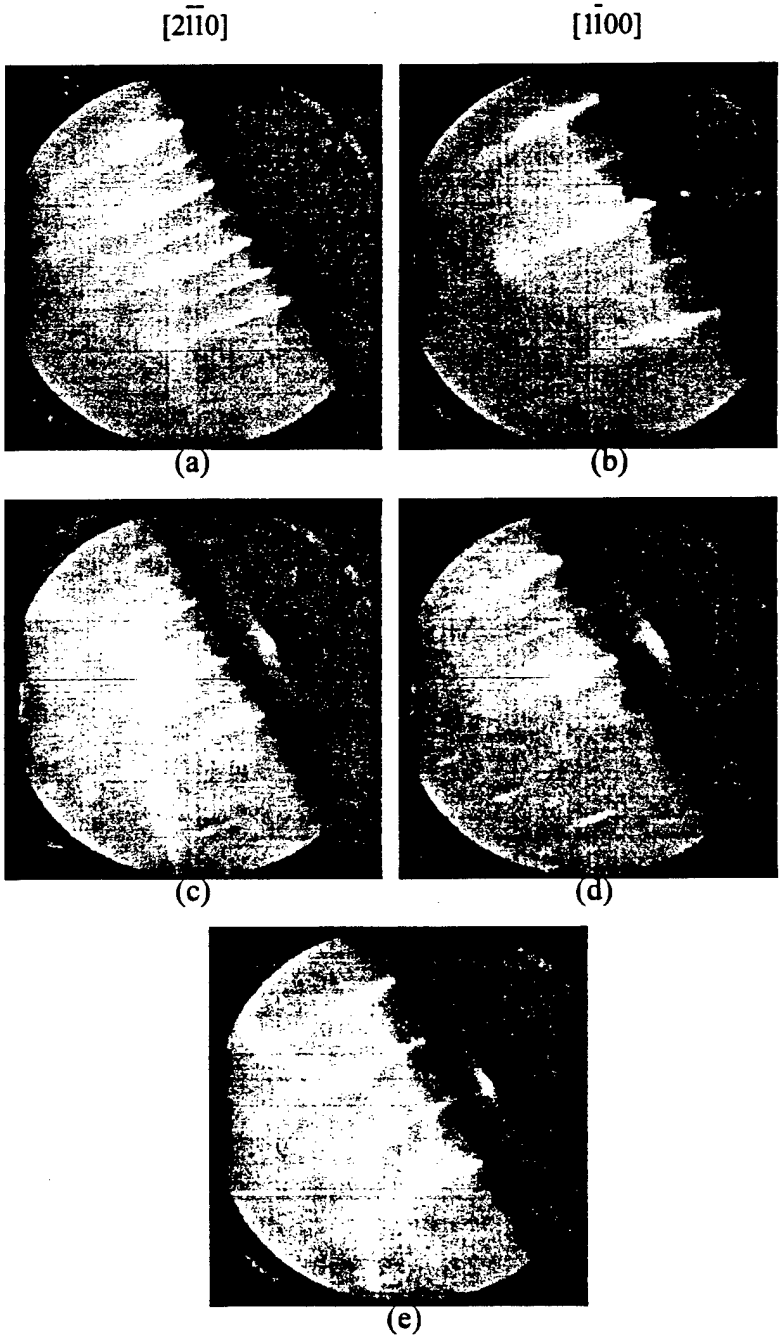


Figure 14. A few representative RHEED patterns taken during this study.

(a) and (b) for typical (2x2) reconstruction, (c) and (d) for (4x4) pattern, and (e) from a typical unreconstructed film.

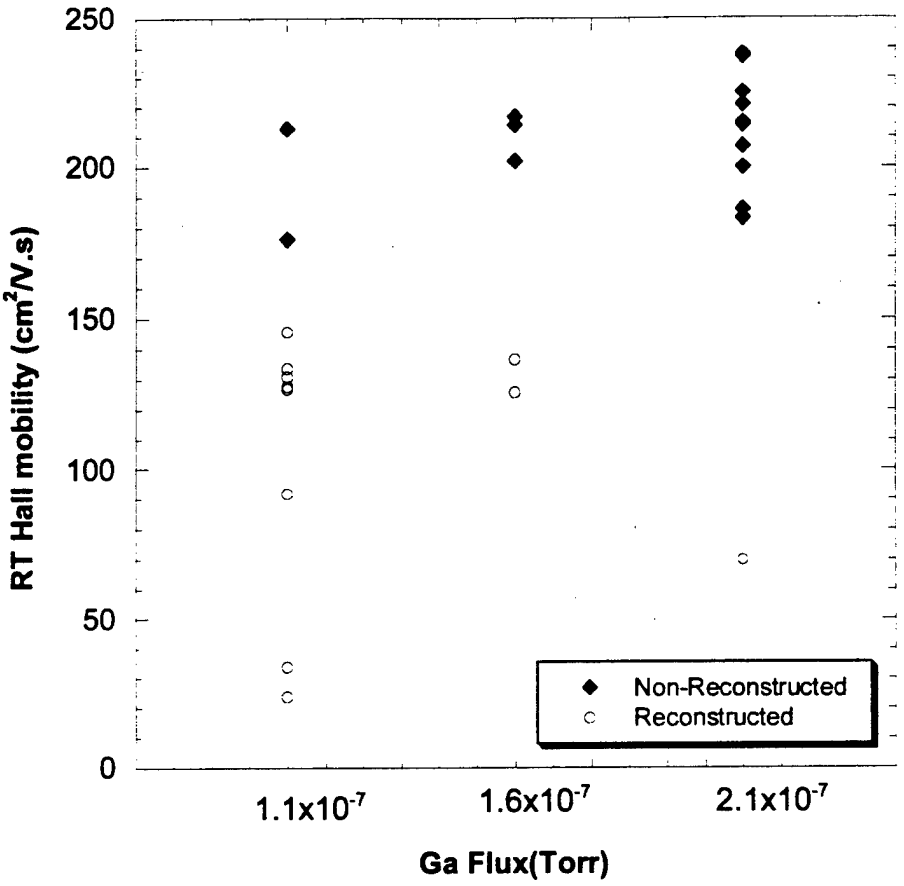


Figure 15 Room temperature Hall mobility vs. Ga flux in BEP with surface reconstructed and non-reconstructed.

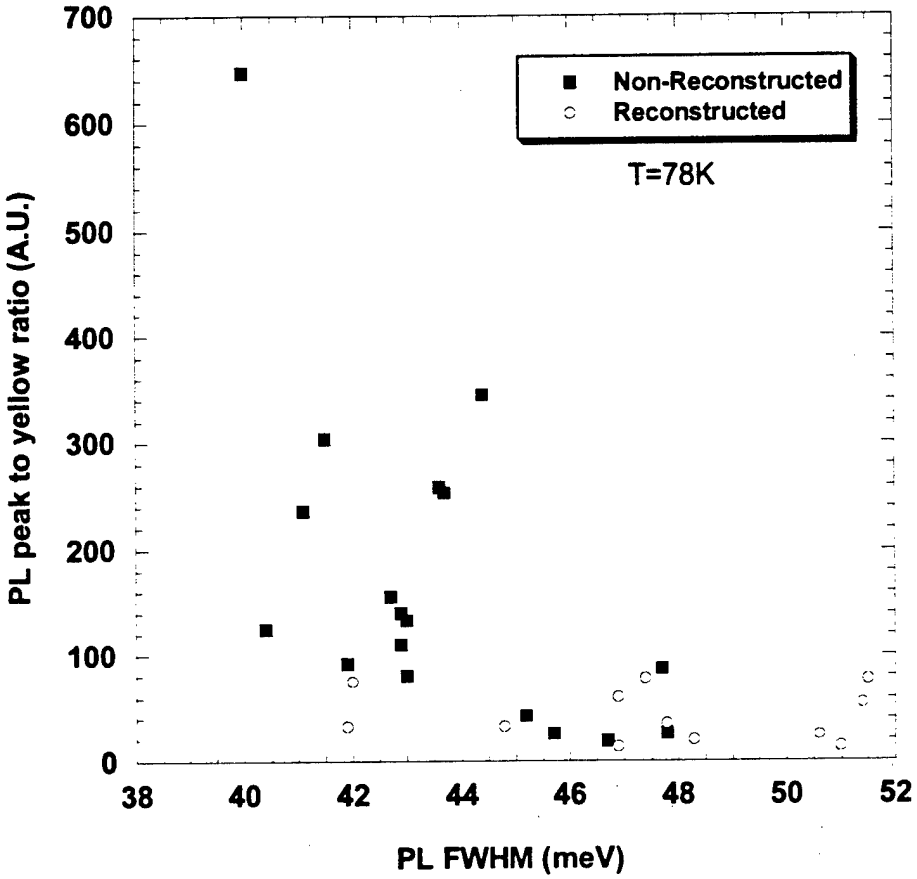


Figure 16 77K photoluminescence characteristics for films with and without surface reconstruction.

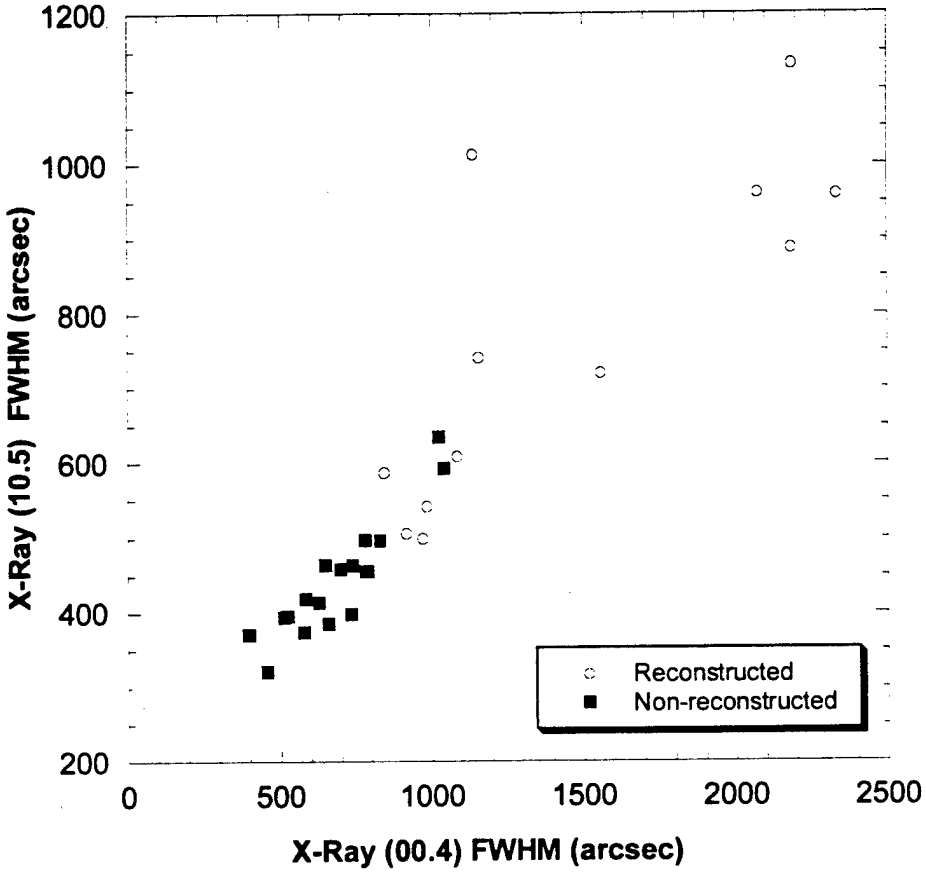


Figure 17 X-Ray DCRC FWHM characteristics.

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